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[54] FINE DETAIL RADIOGRAPHIC ELEMENTS AND EXPOSURE METHOD

[75] Inventor: **Albert L. Van Stappen**, Rumson, N.J.

[73] Assignee: **E. I. Du Pont de Nemours and Company**, Wilmington, Del.

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[58] **Field of Search** 250/475, 480, 482; 96/82

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Primary Examiner—James W. Lawrence
Assistant Examiner—C. E. Church

[57] **ABSTRACT**

A novel film/screen combination particularly useful for the radiographic examination of the breast (e.g. mammography), body-section radiography (e.g. tomography), industrial radiography, and for radiographic examination of the extremities is disclosed comprising a film support carrying a single layer of a fine grain silver halide emulsion in contact with an X-ray intensifying screen and having an antihalation backing coated on the opposite side of the film support. The emulsion having particular speed and grain characteristics combined with a fluorescent X-ray intensifying screen of a particular speed, yields an excellent image free of quantum mottle, or screen noise at low exposure.

8 Claims, No Drawings

FINE DETAIL RADIOGRAPHIC ELEMENTS AND EXPOSURE METHOD

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a novel combination of X-ray film and X-ray intensifying screen suitable for use in X-ray examinations such as those used in mammography, tomography, for extremities and industrial evaluations.

2. Description of the Prior Art

Radiographic examination procedures are now widely used for diagnosis of medical abnormalities and to detect tumors. Where the definition of the abnormality is high compared to the surrounding tissue, high speed medical X-ray film can be used in conjunction with a fluorescent X-ray intensifying screen to detect the abnormality with the lowest amount of patient exposure to X-rays. In the fleshy parts of the body, where the definition of the abnormality is lower, the use of screens in conjunction with an increase in X-ray dosage is not usually successful in locating the abnormality due to side effects caused by the intensifying screen such as "quantum mottle" or "screen noise". These effects are discussed by Sturm et al. in *The Am. Journal of Roent. and Rad. Therapy*, Vol. 62, No. 5 pp. 617–634, Nov. 1949 and by Cleare et al in *Radiology*, Vol. 88, No. 1, pp. 168–174, July 1962. These effects are particularly noticeable when trying to detect tumors and cancerous growths by X-ray examination of the adult female breast. This procedure is known as mammography. In order to properly diagnose suspected growths, it is customary to use relatively thick industrial-type X-ray film without a screen and to increase the X-ray output in order to cause exposure on the film. This technique has been well taught by Leborgne in *The Am. J. of Roent. and Rad. Therapy*, Vol. 65, No. 1, pp. 1–11, January 1951 and by Egan in *Radiology*, Vol. 75, pp. 894–906, Dec. 1960. Exposure times up to 6 seconds at 300Ma, 22–28kVp at a distance of about 30–40 inches are required as reported therein. With six exposures, the total dosage is over 10,000 Mas as compared with about 2 Mas for a normal chest X-ray. In addition, the patient may move during the long exposure time destroying the results and requiring the procedure to be repeated, thus doubling an already dangerously high dosage. Even under the best conditions, involuntary movement of the patient due to heart beats, etc. during the long exposure time causes unsharpness of the image and valuable radiographic information may be lost. Additionally, the industrial-type, thick X-ray film presently used for mammography cannot be processed in the machines at the same time and temperature as is normally used for medical X-ray (i.e., fast access, 90 seconds processing time). Changing machine conditions is tedious and cannot be reproducibly done from time to time. Thus, they are sometimes processed in tanks or trays. The modern hospital may not be fully equipped with a photographic dark room and much additional valuable time may be lost. Some industrial X-ray films are now designed for fast access machine processing but still present the disadvantages of exposure times and patient dosage mentioned above.

Another procedure now used in X-ray diagnostic work is known as "tomography" or "body-section radiography". This technique is well described by Selman in *The Fundamentals of X-ray and Radium Physics*, Charles C. Thomas, Publisher, 1967, pp. 344–351. Some areas of the human body are difficult to radiograph without confusing shadows of superimposed structures. The body-section radiographic technique uses a moving radiographic X-ray source and a photographic film moving in the opposite direction. Several films are exposed in this manner, and at one point, the desired structure will appear the sharpest. Photographic films used for this radiographic analysis are usually of the high speed type, and the emulsion is coated on both sides of the support. Thus, the resulting silver images are separated in distance by the thickness of the support layer. When the incident x-radiation used to expose the image is perpendicular to the film, both image areas will be directly superimposed and the image will be sharp. In body-section radiography, however, the incident light is sometimes not perpendicular to the film surface, and the image on one side of the film support will be slightly out of line from that on the other side resulting in an un-sharp picture.

Yet another application for X-ray films is in the industrial field. Here, metal parts such as aircraft wings, steel girders for bridges, etc. are evaluated for internal flaws and stress points by exposing the desired area using standard, thick, double-coated industrial X-ray film. Since metal has a higher resistance to the penetration of the x-radiation, higher energy or X-ray output is needed. Standard industrial X-ray film is normally used without screens, since the films are coated with a relatively thick layer of sensitive silver halide, and the use of screens would produce poor image sharpness which would severely interfere with this particular analysis. These films, of course, cannot be processed in the machines under so-called fast access conditions (i.e. 90 seconds processing time) and thus rapid evaluation of results is not possible.

It has been found according to the present invention that radiation exposure can be dramatically reduced without sacrificing image contrast, using an optimal combination of a fine grain silver halide emulsion and intensifying screen. Additional advantages include reduction in motion unsharpness, rapid film processability and conservation of silver.

SUMMARY OF THE INVENTION

The invention relates to a radiographic element comprising a film support with a single silver halide emulsion layer and an antihalation layer, said silver halide emulsion having an average silver halide grain size of 0.4 to 1.2 microns, and an X-ray intensifying screen in contact therewith, said emulsion layer and screen having a speed factor between 2.5 and 4.5. The invention also includes the process of exposing said radiographic element to X-radiation.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The X-ray screen is preferably held in intimate contact with the silver halide X-ray film by an evacuated vacuum cassette with flexible sides, e.g., a plastic bag, as is well known in the art. The film-screen combination has characteristics particularly designed, so that the ultimate in photographic speed is balanced with the diagnostic information obtainable at the lowest possible X-ray dosage. This combination yields a product completely compatible with current X-ray laboratory processing systems and yet able to match all current diagnostic information at much lower X-ray dosage than that employed with industrial X-ray film when used, for example, in mammography.

The photographic film useful herein is comprised of a support having a single photographic gelatino silver halide emulsion coated thereon having the average silver halide grain size between 0.4 to 1.2 microns. Preferably, this emulsion is comprised mainly of silver bromide grains with small amounts (about 1 to 4 mole percent) of silver iodide. The emulsion is preferably sensitive mainly to blue light so as to be particularly useful when used with an X-ray intensifying or fluorescent screen. The contrast in the toe portion of the H&D curve of the emulsion will be at least 2.5 in the region of D = 0.2 to 2.0. The emulsion is preferably prepared with a controlled amount of binder, so that the resulting product, when coated on a suitable support (i.e., polyethylene terephthalate) is processable in rapid access machines as taught, for example, by Barnes in U.S. Pat. No. 3,545,971. Preferably, the film support will also contain a blue dye incorporated therein as taught, for example, by Johnes et al in British patent appln. Ser. No. 1,196,707, 10/**28/70.** A gelatino layer containing an antihalation dye dischargeable in the processing fluids, is preferably coated on the films support opposite to that side bearing the silver halide emulsion layer. It may, however, be on the same side as the emulsion layer. Such antihalation layers are used to prevent reflected light and are well known in the art. A gelatin protective top coat is applied as a contigous layer to the silver halide bearing stratum.

The X-ray intensifying screen used in conjunction with the photographic film described above is preferably prepared by dispersing a suitable phosphor (e.g. calcium tungstate) in a binder such as chlorosulfonated polyethylene, n-butyl acetate, or cellulose acetate, etc., using a solvent such as ethyl alcohol. Other adjuvants such as wetting agents, etc., may also be incorporated therein and the resulting dispersed phosphor may be coated over a suitable reflective layer (e.g. TiO_2) which has been applied to a suitable sheet support as well known to those skilled in the art. A protective top coat, e.g. cellulose acetate, may also be applied thereon so as to provide suitable protective qualities to the finished screen and prevent subsequent damage during handling. The reflective underlayers used in screens of this invention may also contain optical brighteners such as the aminocoumarins of Patten as taught in British Pat. No. 999,780, July 28, 1965. Filter dyes which screen out unwanted radiation as known to the skilled artisan may also be used in the phosphor layer or in the protective top coat coated thereon. In place of the calcium tungstate described above there may be substituted known phosphors such as zinc sulfide, zinc oxide and calcium silicate, zinc phosphate, alkali metal halides, cadmium sulfide, cadmium selenide, zinc selenide, zinc telluride, cadmium telluride, cadmium tungstate, magnesium fluoride, zinc fluoride, strontium sulfide, zinc sulfide, barium lead sulfate, cadmium barium fluoride, gadalonium oxysulfide, lanthanium oxysulfide and mixtures thereof. Phosphors whose efficiency has been increased by the addition of doping agents, e.g., europium and terbium may also be used. The term "phosphors", as used herein, denotes any suitable material that will luminesce upon exposure to X-rays, such as those set forth above.

The screens used with the single emulsion layer photographic films of this invention should possess a certain speed factor. This factor is determined by exposing the film to an X-ray source with and without a screen through a step wedge, developing the exposed film and plotting the resulting sensitometric curves. From these curves, the exposure required to obtain a film density of 1.00 is determined, and the speed factor of the x-ray screen is determined from the following formula:

$$\text{Speed Factor (SF)} = \frac{\log_{10} E_2 - \log_{10} E_1}{.301}$$

where

E_1 = exposure required to give unit film density with screen

E_2 = exposure required to give unit film density without screen

Within the ambit of this invention and the above definition, the screen-film combinations of this invention should have a Speed Factor of between 2.5 and 4.5 and, most preferably, between 3.0 and 4.2, where E is in milliampere seconds (Mas) at a given distance (e.g. 40 inches) and kilovolts peak (e.g. 40 kVp), using, e.g. a 1½ mm aluminum filter. If the speed factor is too high, the granularity of the resulting film used therewith will appear too high, and the fine detail needed in any radiographic examination would be strongly reduced. If the speed factor is too low, increased exposure is necessary which is dangerous to the patient's health as described previously.

Yet another method of expressing the screen speed is to define the energy necessary, in terms of x-ray output, to produce a desired degree of blackening of the photographic film used therewith. For example, with the screens of this invention, used with the films of this invention, and with the x-ray generator set at 40 kilovolts peak (kVp) and 2 milliamperes tube current, one places the film-screen combination at a distance of 40 inches. Exposures are made at these conditions varying the time, and the density of the developed film therefrom is measured. To produce a density of 1.0 on the films of this invention with the above screens requires about 4–20 milliampere seconds (Mas) using a single phase generator through a 1½mm aluminum filter. The most preferred film-screen combinations produce a density of 1.0 with about 6–10 Mas.

The quality of the image which can be produced by the screen/film combination described is such that calcifications in the female breast as small as 0.1 mm can be resolved by the system. One method for demonstrating the ability of a photographic system to resolve small objects is to measure the sine or square wave response, or as it is now called, the Modulation Transfer Function (MTF). The procedure for measuring this response is taught by Perrin in J. Soc. Motion Picture and Television Engrs., 69 151 (1960) and Perrin, ibid, 259 (1960) and its use in X-ray film/screen combinations is described by Coltman in J. Opt. Soc. Am., 44 468 (1954) and by Rossman in J. Opt. Soc. Am., 52, 774 (1962). The spatial frequency of a test pattern produced by the system is measured in cycles/mm and the results shown on an oscilloscope. Typical MTF values for the film/screen combination of this invention are shown below:

MTF Value (%)	Frequency (Cycles/mm)
100	0
100	1
75	2
61	3
50	4
41	5
33	6
26	7
21	8
18	9
15	10
13	11
12	12
10	13
9	14
8	15
7	16
1	17

In order to produce a properly defined image within the scope of this invention, the film/screen combination should have a MTF value no less than 90% of these values at any frequency. For example, at 2 cycles/mm frequency, the MTF value should be at least 67.5 (i.e., 90% of 75).

As described above, emulsions useful within the ambit of this invention preferably are composed mainly of silver bromide with small amounts of silver iodide. However, emulsions containing other halides (e.g. chloride or mixtures of bromide-chloride-iodide) may also be used if properly prepared and sensitized. These emulsions are usually brought to their optimum sensitivity as regards speed, fog and contrast, with, for example, gold and sulfur as taught in U.S. Pat. Nos. 1,574,944; 1,623,499; 2,410,699; 2,399,083; 2,448,060 and 2,597,915 as well as the reducing agents of U.S. Pat. No. 2,487,850. Other sensitizing agents and speed increasing adjuvants well-known to those skilled in the art may also be advantageously employed herein. After sensitization is complete, other adjuvants (e.g. wetting and coating aids, hardeners, antifoggant and stabilizers, etc.) may also be added. The emulsion may be coated on any suitable film support (i.e. polyethylene terephthalate prepared and subbed as described in Alles U.S. Pat. No. 2,779,684, Example IV) by any of the means disclosed in the prior art (i.e. skim, air-knife, bar or falling film coating techniques). Other suitable supports include glass, cellulose acetate, cellulose nitrate and other synthetic film forming resins or polymers (e.g. polyester, polyamides, polystyrene, etc.). The film elements may also contain anti-curling backing layers and said backing layers may contain other adjuvants such as antistatic or slip agents, antihalation dyes, etc., in fact it is so preferred. The use of antihalation dyes within the backing layer of the preferred element enhances the image quality by preventing light reflection. The antihalation layer may also be coated under the emulsion layer as known to those skilled in the art.

The emulsions of this invention may contain any of the well-known binding agents such as gelatin, albumin, agar-agar, gum arabic, dextran, alginic acid, polyvinyl alcohol, polyvinyl pyrrolidone, cellulose ether, partially hydrolysed cellulose acetate, alkyl acrylate polymers, the modified and hydrolysed gelatin of Rakoczy, U.S. Pat. No. 3,778,278, or mixtures of two or more of those described above, may be used equally as well. One only needs to balance the sensitivity of the emulsion used and the inherent grain size with the speed range of the intensifying screen described previously to achieve the desired effect. This fact could not have been predicted from the prior art, since it was taught that the use of intensifying screens with photographic films increased the "noise" level so that fine details would be lost. Contrary to this teaching, it has now been found that the balance of emulsion and screen characteristics according to the invention will produce an element with good speed (i.e. lower patient dosage) and effectively record the necessary radiographic information. Additionally, by proper selection of binder and binder/silver halide ratio of the single emulsion layer one may achieve an element that may be processable in the automatic developing machines now widely used in the art. The use of a single layer also prevents exposure from two sides of the film from occurring. This is known as "print-through" and results from an exposure with double coated film and two x-ray screens. Light emitted from one screen should expose the emulsion only on the side nearest the screen. In point of fact, some light returns through the base and exposes emulsion on the reverse side resulting in a blurred image. This is highly undesirable and cannot be tolerated expecially in the X-ray evaluations previously described. The novel system of the invention prevents print-through by providing only a single side coated emulsion and one screen. Since the speed of the system is high enough to use short exposure times, involuntary movement of the patient, which also results in image blurring, is not a problem. All of these advantages were a distinct need in the fields of mammography and of radiological evaluation of extremities.

This invention will now be illustrated by, but is not limited to, the following examples:

EXAMPLE 1

A silver halide emulsion comprised of about 98 mole percent silver bromide and about 2 mole percent silver iodide was prepared in the manner known to those skilled in the art. The silver halide mean grain size was kept at about 1.0 micron by carefully controlling the variables of rate of addition of the silver nitrate to the ammoniacal halide solution and the ripening time and temperature. During the preparation procedure, only a small amount of inert bone gelatin was present (about 20 g./1.5 moles of silver halide). The silver halide precipitate in this small amount of gelatin was then redispersed by vigorous stirring in water and gelatin (about 90 g. gelatin/1.5 moles of silver halide) and the pH adjusted to 6.5 ± 0.1. The emulsion was then brought to its optimum sensitivity by digestion at a temperature of about 140°F with gold and sulfur sensitizers. The usual wetting agents, coating aids, antifoggers and emulsion hardeners were added thereto. All of these steps, adjuvants and methods of preparation are well-known to those skilled in the art of emulsion making and any number of sensitizers and other adjuvants can be used with equivalent results. The emulsion was coated on polyethylene terephthalate film base containing a blue dye which imparts a blue tint to the base as described in British patent application Ser. No. 1,196,707. The film base had been first coated on both sides with a vinylidene chloride-alkyl acrylate/itaconic acid copolymer mixed with an alkyl acrylate and/or methacrylic polymer as described by Rawlins in U.S. Pat. No. 3,433,950 over which had been coated on both sides a thin anchoring substratum of gelatin (0.5 mg/dm^2l). The dried emulsion coated thereon had a coating weight of about 95mg of silver halide/dm^2 and was overcoated with a thin protective layer of hardened gelatin of about 10 mg gel/dm^2. Coated on the opposite side of the film support was a gelatin anticurling and antihalation layer containing about 60 mg gel/dm^2 and antihalation dyes which absorb principally between about 370 nm and 550 nm and are dischargeable in the processing fluids used to develop and fix the photographic image later applied to the silver halide layer.

An X-ray intensifying screen was prepared by first coating a TiO_2 reflective layer comprised of TiO_2 dispersed in a chlorosulfonated polyethylene binder solution (16% chlorosulfonated polyethylene in, for example, n-butyl acetate/mixed petroleum naphtha solution) on 0.01 inch thick biaxially oriented polyethylene terephthalate sheet to a wet thickness of about 0.001 inch. A phosphor suspension comprising 6600 g. of $CaWO_4$ phosphor in a 13.3% polyvinyl butyrate binder solution (n-butyl acetate/n-propanol solvent) was ball-milled for about 16 hours and cast over the TiO_2 layer at a wet thickness of about 0.013 inches, dried and overcoated with a protective layer of cellulose acetate dispersed in acetone to which had been added 8 mls. of a dye solution/1000 g. of the cellulose acetate solution. The dye solution comprised a yellow dye (C.I. Solvent Yellow No. 47) in alcohol, 25g. dye/805 ml. alcohol. The protective coat was applied to the dried phosphor layer at a wet coating thickness of about 0.0013 inches and subsequently also dried.

A sample of the film prepared above was placed in contact with the screen described, with the protective layer of said screen in contact with the emulsion layer of the film element, and this combination, which had a speed factor of 3.1, was placed in a vacuum cassette and evacuated to insure good film/screen contact. This cassette was then placed with the side containing the antihalation layer facing an X-ray source, and a "phantom" which simulates the female breast plus a hand (to test for extremities) phantom were placed thereon. The breast phantom included fine aluminum wires incorporated therein to simulate fine calcium deposits, which may be indicative of early carcinoma of the breast (i.e., during a mammographic examination for breast cancer), a cellulose capsule to simulate coarse, opaque lumps and a gelatin capsule to simulate coarse, transparent lumps. The hand phantom contained cubes of a calcium compound to simulate bone structure. This phantom is made by Minnesota Mining and Manufacturing Co. and is standard for such simulated evaluations. The X-ray source was 40 inches away from the film and was operated at 40 kVp. At a tube current of 100 Ma the element was given a 0.6 second exposure (60 Mas) whereupon an excellent image was obtained when the film was developed in a Kodak M-6 automatic processor at 33°C. containing a standard p-N-methylamino hydrosulfate/hydroquinone X-ray developer, with the total processing time of 90 seconds (develop, fix, wash and dry). In comparison, an industrial X-ray film (300 mg. AgBr/dm^2) without an intensifying screen, required an exposure up to 600 Mas to achieve the same image quality and additionally could not be machine processed under fast access conditions. Also, because the film/screen combination of this invention achieves the required contrast at lower film densities (known as the "toe" portion of the H&D curve) the image could be evaluated on a normal view box while the industrial film (presently used for mammographic work) needed a high intensity viewing light source to see the fine details. The use of high intensity viewing light is unpleasant for the viewer. If instead of the phantom a live, human subject had been employed, due to the increased exposure time necessary with the non-screened element, involuntary patient movement (heart beat, etc.) would have reduced the sharpness of this element so othat the screened version would have had diagnostic value superior to the non-screened element. On the other hand, using a screen or screens with the double coated X-ray element would result in poor definition and lower image clarity due to mottle and print-through.

EXAMPLE 2

Several film/screen combinations were evaluated under the exposure-development conditions described in Example 1, and the results are shown below where a low value indicates increased diagnostic information and a high number indicates unsharpness or mottle:

Film	Screen	Exposure Required (Mas)	Image Evaluation
Industrial X-ray (double coated)	None	600	1
Same as Ex. 1	Screen from Ex. 1, Speed Factor = 3.1	60	1
Same as Ex. 1 but emul. coated ½ on each side	2 screens of Ex. 1	50	3–4
Same as Ex. 1	$BaPbSO_4$ (Medium Speed) Screen Speed Factor = 5.8	30	4
Same as Ex. 1	$BaPbSO_4$ (Low Speed, High Detail) Screen Speed Factor = 0.6	80	1–2
Commercial Double Coated High Speed Medical X-ray	$BaPbSO_4$ (Medium Speed) Screen Speed Factor = 5.8	10–20	4
Commercial Double Coated High Speed Medical X-ray	Screen From Ex. 1 Speed Factor = 5.1	10–20	4

NOTE: 1–2 appear acceptable while 3–4 are considered unacceptable.

This example demonstrates that the specific screen-film combination of this invention is required to produce the best results at the lowest patient exposure. Faster, more grainy film (e.g., commercial high speed medical X-ray film) will not give quality results. Screens with higher or lower speed factors also result in a poor image while many non-screen systems require **10** fold exposure and cannot be machine processed using fast access conditions.

EXAMPLE 3

This example illustrates the calculation of speed factor for film-screen combinations of the invention. A sheet of film of this invention is placed about 40 inches from a suitable X-ray source operated at, for example, 40 kilovolts peak (kVp) and 200 milliamperes (Ma) tube current through a 1½ mm aluminum filter. Stepped exposures are made on this film using a lead shield to cover all but about a ¾ inch strip across the cassette containing the film. This lead is moved after each exposure to present a new, unexposed strip of film to the radiation. At least 5 exposures are made varying the exposure time so that the first strip exposed had a total of 8/**10** seconds, the second 4/**10** seconds, the third 2/**10** seconds, the fourth 1/**10** seconds and the fifth 1/**20** seconds giving a resulting exposure of 160, **80, 40 20** and 10 milliamp-seconds (Mas). The exposed film was developed as described above and the densities of the various exposures were read and plotted on $\log_{10}$ paper with the "X" axis being exposure and the "Y" axis being density. A curve was drawn through these points and the exposure necessary to get D = 1.00 was read from this curve. In this case, it was 64 Mas. Thus steps were repeated for a second sheet of film but including the screen of this invention. The tube current was lowered to 25 Ma and the exposure times were **4/10, 2/10, 1/10, 1/20** and 1/**40** seconds giving 10, 5, **2.5, 1.25** and 0.625 Mas respectively. D = 1.00 was reached at 8 Mas. The speed factor was calculated from the formula

$$SF = \frac{\log_{10} E_2 - \log_{10} E_1}{.301}$$

where E_2 = exposure required to give unit film density without a screen = 64 Mas

and E_1 = exposure required to give unit film density with a screen = 8 Mas

$$SF = \frac{\log_{10} 64 - \log_{10} 8}{.301} = 3.1$$

What is claimed is:

1. A radiographic element comprising a film support bearing a single silver halide emulsion layer and an antihalation layer, said silver halide emulsion having an average silver halide grain size of 0.4 to 1.2 microns, and an X-ray intensifying screen in contact with said emulsion layer, said emulsion layer and screen having a speed factor between 2.5 and 4.5.

2. A radiographic element of claim **1** wherein the silver halide average grain size is about one micron.

3. A radiographic element of claim **1** wherein the speed factor is between 3.0 and 4.2.

4. A radiographic element of claim **1** wherein said emulsion layer and screen are held in intimate contact in an evacuated vacuum cassette.

5. A radiographic element of claim **1** wherein said element has a modulation transfer function of at least 67.5 at 2 cycles per mm.

6. A method of making fine detail radiographs with low X-ray exposure comprising exposing the element of claim **1** to X-radiation.

7. A method according to claim **6** of making mammographic radiographs with fine detail at low exposure levels comprising exposing said element to X-radiation through a human female breast.

8. A method of claim **6** wherein exposing said element to X-rays in an amount between 4 to 20 milliampere seconds at a distance of 40 inches is capable of producing a developed image density of 1.0 on said silver halide emulsion layer.

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